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LOW TEMPERATURE TENSILE PROPERTIES
OF ZONE REFINED NIOBIUM (COLUMBIUM)

A THESIS SUBMITTED IN

1962

BY

JOHN F. WELLINGS

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LOW TEMPERATURE TENSILE PROPERTIES OF
ZONE REFINED NIOBIUM (COLUMBIUM)

John F. Wellings
/

A Thesis
in Metallurgical Engineering
Presented to the Faculty of the
School of Metallurgical Engineering
University of Pennsylvania, in partial fulfillment
of the requirements for the degree of
Master of Science

1962

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TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	iv
INTRODUCTION	1
EXPERIMENTAL PROCEDURES	7
Outline of Investigation	
Description of Apparatus	
Experimental Techniques	
RESULTS AND DISCUSSION	20
Purity of Zone Melted Niobium (Columbium)	
Resistivity Measurements	
Orientation of Single Crystals	
Calculation of Resolved Shear Stress for	
Single Crystals	
Results of Tensile Tests	
Twinning	
Slip Lines	
Yield Point Phenomena	
Fracture	
Temperature Dependence of the Flow Stress	
Temperature Dependence of the Yield Stress	
CONCLUSIONS	36
BIBLIOGRAPHY	37
TABLES	40
FIGURES	48

LIST OF TABLES

Table	Page
1. Analyses of Zone Melted Niobium (Columbium) . .	40
2. Resistivity of Zone Melted Niobium (Columbium). .	41
3. Orientation Factors of Single Crystals	43
4. Resolved Shear Stresses at 0.05%	44
5. Tensile Testing Scheme	45
6. Change in Flow Stress	46
7. Polycrystalline Yield Stress at 0.05% Plastic Strain	47

INTRODUCTION

Because interest in body centered cubic metals other than iron is relatively recent, the first theories on the mechanical behavior of this group of metals were based on work done with iron. Cottrell and Bilby (1) reasoned that the yield point of iron exhibits a strong temperature dependence at low temperatures because impurity atoms are attracted to dislocations, thereby lowering the strain energy of the system. In effect then, each dislocation is surrounded by a cloud of impurity atoms which tend to lock it in place. In order to move, the dislocation requires a much larger stress than if it were free of its interstitial atmosphere. Thus, when a stress is applied, the dislocation must either break away from the impurity atoms which are blocking its movement or carry them with it. Since the movement of the impurity atoms is diffusion-controlled (very slow), the applied stress builds up until it is high enough to break the dislocation away from its interstitial atmosphere. The free dislocation begins moving and interacts with other dislocations, freeing them and resulting in a catastrophic process which, in turn, propagates Luders bands. This was thought to explain, completely, the

(1) A. H. Cottrell, B. A. Bilby, Proceedings of the Physical Society, vol. A62, 49 (1949).

yielding process in α -iron. Later experimental work with iron seemed to agree with this theory.

Other work on polycrystalline iron concentrated on the effect of grain size on the lower yield stress. It was found that the lower yield stress, σ_{LY} , varies according to the equation:

$$\sigma_{LY} = \sigma_i + K_y d^{-\frac{1}{2}} \quad (1)$$

Where σ_i is a lattice friction stress which impedes dislocation motion, d is the grain diameter, and K_y is a constant related to the stress, σ_D , which is required to free locked dislocations. (1, 2) Interpretation of this equation by Cottrell (3) and Petch (4) explained that dislocations present in a crystal can probably move at a stress much smaller than the yield stress until they reach a grain boundary. On glide planes which contain a Frank-Read source, multiplication will take place until interactions between dislocations within the accumulation at the grain boundary cause the multiplication to stop for the value of stress being applied at that time. These accumulations, or pile-ups, then remain until the stress fields ahead of them are able to operate dislocation sources in adjacent grains.

(1) N. J. Petch, Journal of the Iron and Steel Institute, vol. 174, 25 (1953).

(2) J. Heslop and N. J. Petch, Philosophical Magazine, vol. 1, 866 (1956).

(3) A. H. Cottrell, AIME Transactions, vol. 212, 192 (1958).

(4) N. J. Petch, Progress in Metal Physics, vol. 5, 1 (1954).

This would mean that δ_1 , the lattice friction stress, impedes the individual dislocations in the pile-ups. Heslop and Petch (1) showed that δ_1 is made up of two parts; $\delta_{11}(T)$, which is dependent only on temperature, and $\delta_{12}(st)$, which is a function of structural imperfections, such as interstitials. This hypothesis again was supported by further experimental work with iron, though it did lead to some doubt as to the dominating role of interstitial atmospheres.

With the increased interest in refractory metals during the last decade, it was natural to assume that they would obey the rules which had been found to govern the mechanical behavior of iron. With the increased testing of the other b.c.c. metals, however, several discrepancies were found. Tests made on niobium (columbium) (2) and molybdenum (3) showed that, for these metals, the frictional stress, δ_1 , and not the unlocking stress, δ_D , is responsible for the temperature dependence of the yield strength. Further tests on iron (4) showed that the lower yield stress, δ_{LY} , the flow stress, δ_{f1} , and the frictional stress, δ_1 , all exhibit the same temperature

(1) J. Heslop, N. J. Petch, Philosophical Magazine, vol. 1, 866 (1956).

(2) M. A. Adams, A. C. Roberts, R. E. Smallman, Acta Metallurgica, vol. 8, 328 (1960).

(3) A. A. Johnson, Philosophical Magazine, vol. 4, 194 (1959).

(4) H. Conrad, G. Schoeck, Acta Metallurgica, vol. 8, 791 (1960).

dependence. This indicates that the unlocking of pinned dislocations (yield), the movement of unpinned dislocations (flow), and the resistance to movement of free dislocations (lattice friction) are all controlled by the same process. Thus, dislocation locking would seem to be ruled out as the controlling influence in the yielding of b.c.c. metals. However, later work by Lawley, Van den Sype, and Maddin (1) shows a definite effect of impurities on the temperature dependence of the yield and flow stress in molybdenum. From this it would seem that at least a portion of the temperature dependence of the mechanical properties in b.c.c. metals is due to impurities.

Along with the vast amount of experimental findings to appear in recent years, several other possible explanations for the temperature dependence have been advanced. Heslop and Petch (2) have suggested that the frictional stress is due to a large Peierls-Nabarro force while Schoeck (3) proposes that thermally activated jogs in screw dislocations are responsible. Johnson (4) has proposed a revised model for deformation and fracture in b.c.c. metals. In it, σ_{LY} represents the lower yield stress of a single crystal. It is dependent on the frictional stress,

(1) A. Lawley, J. Van den Sype, R. Maddin, accepted for publication in AIME Transactions (1962).

(2) Heslop and Petch, op. cit.

(3) G. Schoeck, Acta Metallurgica, vol. 9, 382 (1961).

(4) Johnson, op. cit., vol. 6, 177 (1961).

σ_1 , which is made up of $\sigma_1(I)$ and $\sigma_1(L)$. $\sigma_1(L)$ is the stress required to propagate a Luders band through a single crystal having no lattice friction stress. Both of these terms may be temperature dependent, since the first may contain a Peierls-Nabarro stress, while the second may contain σ_D , the unlocking stress. Thus, the yield stress for a b.c.c. single crystal would be given by:

$$\sigma_{LY} = \sigma_1(I) + \sigma_1(L) \quad (2)$$

For a polycrystal, the equation becomes

$$\sigma_{LY} = \sigma_1(I) + \sigma_1(L) + K_y d^{-\frac{1}{2}} \quad (3)$$

where K_y now represents the effect that grain boundaries have on propagating the Luders band rather than the value of the unlocking stress as it did in equation (1). Still another explanation is that offered by Conrad (1) and discussed by Low and Guard (2) and Johnston and Gilman. (3) It concerns the sudden generation of large numbers of dislocations by the double cross-slip mechanism of Koehler (4) and Orowan. (5) This generation takes place at the lower yield point to produce a Luders band and the same mechanism spreads the band front during plastic flow. In this model,

(1) H. Conrad, Journal of the Iron and Steel Institute, vol. 198, 364 (1961).

(2) J. R. Low, R. W. Guard, Acta Metallurgica, vol. 7, 171 (1959).

(3) W. G. Johnston, J. J. Gilman, Journal of Applied Physics, vol. 30, 129 (1959).

(4) J. S. Koehler, Physical Review, vol. 86, 52 (1952).

(5) E. Orowan, Dislocations in Metals, AIME, New York, 103 (1954).

the motion of free dislocations is responsible and the rate-controlling process is assumed to be the overcoming of the Peierls-Nabarro stress.

Each of the models mentioned previously has been supported and disputed by experimental evidence from many tests on b.c.c. metals with the result that a clear-cut picture of the deformation of these metals has not yet been found. It is the purpose of the present work to add weight to the arguments for one of these models, in the hope that a clearer explanation will result.

EXPERIMENTAL PROCEDURES

Outline of Investigation

The niobium (columbium) used in this investigation was in the form of arc-cast, swaged rods approximately 0.073 inches in diameter. It was zone melted in an electron beam melting apparatus as described by Davis et al. (1) for refractory metals. The apparatus was constructed by A. Lawley (2) in the University of Pennsylvania School of Metallurgical Engineering. Single crystals resulted from the zone melting operation. Orientation of the crystal axis was determined by the Laue back-reflection method. Where polycrystalline material was to be used, the single crystal rods were swaged and annealed in the zone melting apparatus. Tensile specimens were cut from the zone melted rods and gage sections were produced using an acid lathe. The specimens were tested in an Instron Testing Machine. Resistivity measurements were made using a standard potentiometer arrangement.

Description of Apparatus

1. Electron Beam Zone Melting Apparatus

The specimen, which is mounted vertically (Fig. 1),

(1) M. Davis, A. Calverley, R. F. Lever, Journal of Scientific Instruments, vol. 34, 142 (1957).

(2) A. Lawley, Electronics, vol. 32, 39 (1959).

acts as the anode and target for the electrons which are produced by a cathode filament made of non-sag tungsten wire 15 mils in diameter. The filament is circular in shape and travels the length of the specimen at a constant speed of about two millimeters per minute. Drive for the filament is produced by a variable speed motor attached directly to a threaded post on which the filament holder rides. The filament is enclosed in a molybdenum block which is surrounded by sheet molybdenum reflectors. These serve to prevent undue scattering of the bombarding electrons. The specimen is held in place by molybdenum spring clips which allow for expansion and contraction during heating and cooling.

A vacuum of at least 5×10^{-6} millimeters of mercury is produced by an oil diffusion pump and rotary pump combination. The melting chamber is protected from possible oil contamination by a liquid-nitrogen-cooled cold trap. The cover for the melting chamber is a twelve inch bell jar. Protection for the operator against implosion is afforded by a plexiglass shield.

As a specimen is melted, gases from impurities and the material itself are given off. These gases ionize and bombard the filament, increasing its temperature, hence, its emission rate. This, in turn, would increase the temperature of the specimen if allowed to continue. In order to regulate the cathode temperature, the emission current is passed through a resistor and the voltage compared to a standard voltage. The value of the difference

between the two is fed to a controller which varies the cathode temperature, thus affording automatic emission control.

2. Acid Lathe

The acid lathe consists of an aluminum framework which supports an axle on which a stainless steel wheel is mounted (Fig. 2a). The wheel is driven by a 25 rpm motor. At the opposite end of the framework, a one rpm synchronous motor is mounted. Its shaft has a drill-press chuck attached to it to hold the specimen to be polished. A polyethylene tray filled with the electropolishing solution is placed beneath the wheel which rotates in a direction opposite to that of the chuck. The solution, which consists of two parts lactic acid, one part sulfuric acid, and one part hydrofluoric acid (1), is drawn up by the moving wheel and forms a film between the wheel and the specimen. A 22 volt dry cell battery is used to produce a voltage drop of 15 to 20 volts across the gap between the specimen and the wheel. A platinum cathode (wheel) is desirable for electro-etching niobium (columbium) but satisfactory, though somewhat slower results were obtained using the stainless steel wheel. Only minor damage to the wheel resulted.

3. Tensile Grips and Frame

The same grips were used for testing at all temperatures. They were hollow, stainless steel cylinders,

(1) W. J. Tegart, The Electrolytic and Chemical Polishing of Metals, Pergamon Press, New York, 68 (1959).

0.75 inch in diameter (Figs. 3 and 4). Hardened, stainless steel inserts with knurled holes fitted inside the cylinders and a threaded cap held the inserts in place. Four set screws in each cylinder provided a means for squeezing the inserts together to hold the tensile specimen. The caps were threaded internally to receive a 1/4 inch diameter extension rod (to the Instron load cell) and another extension to a cylindrical, slotted block which was attached to two links of chain by means of a pin. The chain acted as a universal joint at the bottom of the tensile train. The bottom link of the chain ran through a slot in the base of a stainless steel framework and was attached by another pin. The framework was attached to the bottom of the Instron crosshead and the whole assembly was immersed in the various temperature baths.

4. Cryostat

For measurements in liquid helium, a stainless steel tube 1.75 inches in diameter was used instead of a large framework (Fig. 4). A plate with a 1/4 inch hole in the center was attached inside the tube 3 inches from the top. The plate contained a set screw which allowed the stainless steel tensile extension to be gripped, thus preventing vertical movement of the tensile train until the test was started. Below the plate, an exhaust port was inserted into the tube to allow the liquid helium which vaporized to escape. Several holes were drilled in the tube to allow free movement of the liquid helium and the bottom was fitted with a slotted base plate to receive the lower link of the

extension chain and a holding pin. A wire screen was placed under the base plate and wired to the tube to prevent the lower elements of the tensile train from falling into the liquid helium dewar when the specimen fractured.

5. Specimen Holder for Resistivity Measurements

The holder used for resistivity measurements consisted of a teflon base which measured $3/4 \times 5 \times 1-1/2$ inches (Fig. 2b). Two stainless steel cylinders $1/2$ inch in diameter were sunk into the base. One cylinder had a hole with a set screw to fasten the lower end of the specimen. The other cylinder was slotted and the slot covered by a spring clip to provide electrical contact but allow for contraction of the specimen. Both cylinders contained attaching screws for current leads. A teflon platform 3 inches in length was built up from the base. This contained two spring-loaded copper knife-edges which were spaced $2-1/2$ inches apart. Both knife-edges contained attaching screws and washers for voltage measurement. An eye was attached to the upper end of the holder to facilitate lowering the holder into the liquid helium.

Experimental Techniques

1. Zone Melting

Niobium (columbium) rods were cut into 4 inch lengths and cleaned with acetone to remove surface dirt. One end of each length was machined flat. The unmachined ends of two lengths were placed in the molybdenum spring clips and the clips were tightened. One clip was left

finger-tight in order to allow for expansion of the rod on heating. If this is not done, the specimen may warp enough to touch the filament, thus short-circuiting the system. The two sections were aligned so that their flat ends were touching and the filament was positioned at the joint. As soon as the necessary vacuum level was attained, the touching ends were welded together. This procedure is necessary in order to further prevent bending of the rod due to relief of strains from previous cold work. It also gives an estimate of the power needed for the following passes. When the parts were welded, the filament was moved to the top of the specimen, the power was adjusted to create a molten zone, and the filament was moved downward at about two millimeters per minute. It was usually necessary to vary the power on the second and succeeding passes, since the first pass resulted in areas of decreased diameter along the rod.

The power required to melt the niobium (columbium) specimens was about 84 watts (2.1 kv and 40 ma), as contrasted with the 280 watts used by Van den Sype (1) for melting molybdenum rods with the same apparatus. This difference was due to the lower melting point of niobium (columbium) and the smaller diameter of the niobium (columbium) rods (0.07 inch to 0.12 inch). Van den Sype also experienced a great deal of vaporization, both from impurities and the molybdenum. This was not the case with

(1) J. Van den Sype, Tensile Properties of Zone Refined Molybdenum, M.S. Thesis, University of Pennsylvania (1961).

the niobium (columbium). Very little outgassing was observed and with the low power being used, it was possible to make six zone passes with one filament. The main trouble which was encountered was that the rods used were too thin to support the molten zone under any conditions other than ideal. There was a very small voltage range (about 0.1 kv) between melting and parting the specimen. Since the length of the molten zone which can be sustained by the surface is dependent on the diameter of the specimen, (1) it is felt that an increase in diameter of the niobium (columbium) rods to 1/8 inch would allow at least ten zone passes to be made with a resulting decrease in impurities.

2. Preparation of Polycrystalline Specimens

The product of zone melting was, in all cases but one, a single crystal which ran the length of the rod. (In Run 23 two large grains resulted, each running about half the length of the rod.) For the tensile tests requiring polycrystalline material, the zone melted rods were swaged with a resulting reduction in area of 18 to 42 %. The specimens were then electropolished lightly in the solution mentioned previously. This rid them, at least partially, of any foreign matter picked up during the swaging operation. The rods were then annealed in the zone melting apparatus under a vacuum of 5×10^{-6} millimeters of mercury for approximately 45 minutes per inch of length at a temperature

(1) W. G. Pfann, Zone Melting, John Wiley and Sons, New York (1958).

of about 1450°C (by optical pyrometer). Samples of each annealed rod were mounted in "Quick Mount," mechanically polished, and etched in a solution of hydrofluoric and nitric acids (1) in equal amounts. The samples were then examined under an optical microscope (magnification of 100) and the grain size was found to vary between 0.2 and 0.54 millimeters in grain diameter for the different specimens.

This procedure was also used in preparing the specimen for resistivity measurements. Since resistivity measurements require a constant specimen diameter, the specimen was swaged and annealed in the as-received state and after one and six zone passes. The grain size varied from 0.2 millimeters for as-received to 0.32 millimeters after six passes. This is considered within limits in view of Kunzler and Wernick's (2) findings that resistivity is not a function of grain size in b.c.c. metals.

3. Preparation of Tensile Specimens

Each zone melted rod was cut into two or three sections depending on the length of the rod. The sections were 1-3/4 to 2 inches long and were numbered according to the zone melting run and their position in relation to the start of the zone melting process. (For example, the top or purest portion of the twentieth run would be R20-Spl.)

(1) B. A. Wilcox, A. W. Brisbane, R. F. Klinger, WADD Technical Report 61-44 (1961).

(2) J. E. Kunzler, J. H. Wernick, AIME Transactions, vol. 212, 856 (1958).

Each section was then carefully aligned in the chuck of the acid lathe with equal amounts of the specimen on either side of the wheel. Any misalignment at this point resulted in an uneven cross-section in the gage length. The acid lathe was started and the specimen-to-wheel distance adjusted to provide a meniscus of electrolyte which extended halfway up the specimen. The battery was connected and the apparatus allowed to run for from eight to fifteen minutes, depending on the desired diameter of the gage section. In most cases, the supply of electrolyte had to be renewed after five to ten minutes. The resulting gage diameters were uniform to within ± 0.005 inch as measured by low power traveling microscope.

The average tensile specimen had a shoulder diameter of 0.06 inch, a gage diameter of 0.04 inch and a gage length of 0.5 inch. Polycrystalline specimens were somewhat smaller due to the swaging. The gage diameter of specimens to be tested in liquid helium was reduced even more to insure fracture in the gage section, as recommended by Van den Sype. (1)

4. Tensile Testing

For all tensile tests the Instron machine was adjusted for a chart speed of five inches per minute, and a constant crosshead speed of 0.01 inch per minute. This corresponds to a strain rate of 3.3×10^{-4} seconds⁻¹.

(1) Van den Sype, op. cit.

The testing temperatures used were 373°K (boiling water), 273°K (water and ice), 196°K (acetone and dry ice), 77°K (liquid nitrogen), and 4.2°K (liquid helium). A thermocouple was wired in place next to the specimen for the temperature range 373°K to 196°K . This made it possible to measure temperature to within $\pm 1.0^{\circ}\text{K}$.

The tensile specimen was mounted in the grips which were clamped in a jig to prevent movement and inadvertent bending of the specimen. The tensile train was then assembled and attached to the instron load cell and the bottom link of the chain was pinned to the steel framework. The crosshead was then lowered until a small amount of slack remained in the tensile train. Some slack was necessary in order to allow for contraction of the tensile train or expansion of the framework, depending on the temperature bath being used. A large dewar flask containing the bath was then secured beneath the crosshead so that the tensile train and platform were immersed. This method was used for all testing temperatures with the exception of 4.2°K .

As discussed by Van den Sype, (1) it is necessary to have a constant dislocation structure, subgrain structure, surface condition and grain size in order to make temperature the only variable in tests of this kind. This was accomplished by using the testing method of Conrad and Schoeck. (2) A specimen at one temperature was pulled to

(1) Ibid.

(2) H. Conrad, G. Schoeck, Acta Metallurgica, vol. 8, 791 (1960).

approximately 0.5% plastic strain. The load was then removed (except for a five pound load to maintain alignment), the temperature bath changed, and the process repeated. This cycle was repeated until fracture of the specimen occurred. Care was taken to allow the temperature of the specimen to stabilize in each new temperature bath and to maintain the alignment load despite expansion and contraction of the specimen and platform.

For tests in liquid helium, the cryostat previously described was used. The specimen was mounted in the grips and the tensile train was assembled in the cryostat. The upper extension was secured in the top plate by means of the set screw and the lower link of the chain was pinned to the base plate. Again, enough slack was left in the train to allow for contraction on cooling. The bottom screen was then wired in place and the whole assembly lowered into acetone. After drying to remove moisture, the cryostat was placed in liquid nitrogen and the temperature allowed to stabilize. Then the cryostat was removed from the nitrogen and lowered slowly into the liquid helium. During this operation, cotton wool was placed around the top of the helium dewar to inhibit the evaporation of helium. Also, the cryostat was turned as it was lowered into the dewar in order to prevent formation of ice between the dewar and cryostat walls. After waiting for equilibrium temperature to be reached, the upper extension was attached to the Instron load cell and the test was started. All

specimens tested at liquid helium temperature were pulled to fracture without changing the temperature bath.

5. Resistivity Measurements

Resistivity measurements were taken in order to determine the relative degree of effectiveness with which the zone melting had been carried out. In order to obtain a value of resistivity from the resistance which is measured, the exact cross sectional area of the specimen must be known, along with the exact distance between measurement contact points. (1) It was decided to swage the specimen after each zone melting treatment to insure a uniform area. In order to insure uniform internal structure, it was decided to recrystallize the specimen and attempt to obtain approximately the same grain size after each annealing.

An as-received niobium (columbium) rod was treated as described previously to produce grains 0.2 millimeters in diameter. After measuring the diameter, the specimen was placed in the holder, making sure that the copper knife-edges and end grips made good contact. Current and measurement leads were attached and the entire assembly was immersed in distilled water and ice. Distilled water was used in making the ice in order to minimize the concentration of conducting impurities in the solution. After allowing time for equilibrium temperature to be reached, resistivity measurements were taken at ten minute intervals until

(1)Kunzler and Wernick, op. cit.

a stable reading could be recorded. The holder was then dipped in acetone and dried to remove moisture and placed in liquid nitrogen. It was then lowered slowly into liquid helium and readings were taken as before. This process was repeated after the specimen had been given one zone pass, swaged and annealed, and again after it had been given six zone passes, swaged, and annealed. The grain size after the second swaging was 0.28 millimeters, and after the third, 0.30 millimeters. After the readings on the six pass specimen, the position of the probes was changed toward the lower end of the rod and a new set of readings in liquid helium and in ice water were taken. This was done to determine whether there were more impurities toward the lower end of the zone melted specimen.

RESULTS AND DISCUSSION

Purity of Zone Melted Niobium (Columbium)

It is possible for zone melting to decrease impurities in a specimen in three ways:

- (a) Removal of dissolved gases by heating under high vacuum;
- (b) Removal of elements which are more volatile than the basic metal by vaporization;
- (c) Movement of impurities to the end of the specimen within the molten zone or true zone refining. (1)

In zone refining, the molten zone is established. As the molten zone moves down the rod, impurities which lower the melting point of the solvent concentration in the freezing solid will be rejected by the freezing solid. (2) Thus, they will remain in the molten zone and be carried down the specimen. If, on the other hand, the solute impurity raises the melting point of the solvent concentration until it is higher than that in the liquid, the liquid will be depleted of solute which will be left behind at the freezing interface. Impurities with relatively low vapor pressures will be decreased by (b) above. The majority of purification by both (a) and (b) occurs on the first pass

(1) Davis et al., op. cit.

(2) Plann, op. cit.

since their action is dependent only on the heating and the vacuum. For this reason, any purification on succeeding passes can be attributed mainly to zone refining.

In contrast to the results found for molybdenum, (1) it is believed that the purification of the niobium (columbium) used in the present work took place mainly by zone refining, as distinguished from zone melting. Whereas the molten molybdenum gave off quantities of vapor which coated the inside of the bell jar, the molten niobium (columbium) and its impurities gave no traces of outgassing, and on only one occasion were sparks seen to issue from the molten zone. These results are in agreement with those of Buehler (2) in his work on niobium (columbium). Furthermore, significant differences in concentration between one and six zone passes were observed in the cases of oxygen, nitrogen, nickel, and zirconium as reported in the spectrographic and gas analyses (Table 1). Others among the impurities may have been reduced by a like amount, but this reduction was not detectable due to the limitations of the analysis. Finally, a distinct difference in resolved shear stress between one and six zone passes shows that a significant amount of purification did take place during the last five passes (Fig. 5).

Results of the analyses showed the as-received material to be 99.33% pure, and the six zone pass material

(1) Lawley et al., op. cit.

(2) E. Buehler, AIME Transactions, vol. 212, 694 (1958).

to be 99.7% pure. This is very low purity by today's standards. However, when it is considered that 95% of the remaining impurities were the b.c.c. metals tantalum and zirconium, which possibly occurred as substitutional rather than interstitial impurities, any error in the results introduced by their presence would seem to be minimized.

Resistivity Measurements

The resistivity of a metal may be thought of as being divided into three parts; (1) chemical, thermal, and physical, so that

$$\rho_{\text{total}} = \rho_{\text{chem}} + \rho_{\text{therm}} + \rho_{\text{phys}} \quad (4)$$

The thermal portion is due to scattering of conducting electrons by lattice vibrations, while the physical part is due to physical defects. The chemical portion is due to the effects of chemical impurities. According to Matthiessen's Rule, (2) the physical and chemical portions are independent of temperature. At the temperature of liquid helium, the thermal portion is considered negligible due to the minor effect of remaining lattice vibrations.

Thus,

$$\rho_{4.2} = \rho_{\text{phys}} + \rho_{\text{chem}} \quad (5)$$

The physical portion may also be considered negligibly small in zone refined materials, so that the resistivity at 4.2°K is a function of impurities only. Therefore, by computing

(1) Kunzler and Wernick, op. cit.

(2) J. S. Koehler, Impurities and Imperfections, ASM, Cleveland, 162 (1955).

a ratio of resistivity in liquid helium to that at 273°K, a good indication of the impurity content will result.

In order to compute the resistivity, a current is measured through a standard resistor. This current is then passed through the specimen and the voltage drop between the contacts is measured. Dividing the voltage by the current gives the resistance of the portion of material through which the current was passed. Then, by applying the formula

$$\rho = \frac{RA}{L} \quad (6)$$

the resistivity may be found. In this formula R is the measured resistance, A is the cross-sectional area of the specimen, and L is the length between contacts. The resistivity ratio, $\rho_{4.2}/\rho_{273}$, is then computed from the values of resistivity at the two temperatures.

The results in Table 2 show that there is a significant drop in the resistivity ratio between the as-received and one-pass specimen, but a much greater drop for the six pass material. The ratio for the six pass specimen near the lower end shows a rise, hence an increase in impurities near the end. Both of these results further indicate that zone refining rather than volatilization and out-gassing is the principle mechanism of purification as stated previously.

Orientation of Single Crystals

The orientations of the axes of the niobium (columbium) single crystals produced by zone melting are shown on the projection of the standard triangle in Figure 6.



Figure 6

Calculation of Resolved Shear Stress for Single Crystals

The stress resolved on a slip plane in the slip direction is given by the equation

$$\sigma = F/A \cos \phi \cos \lambda \quad (7)$$

Where F is the applied load, A is the original cross-sectional area, ϕ is the angle between the stress axis and the normal to the slip plane, and λ is the angle between the stress axis and the slip direction. (1) Thus, the multiple, $\cos \phi \cos \lambda$, called the orientation factor, may be calculated if the slip plane, slip direction, and orientation of the stress axis are known. For b.c.c. metals, the close-packed direction, 111 , is the slip direction, while the slip plane may be a $\{110\}$, a $\{112\}$, or a $\{123\}$. (2) The slip system which operates is assumed dependent on the orientation of the crystal with respect to the stress axis

(1) N. P. Allen, B. E. Hopkins, J. E. McLenna, Proceedings of the Royal Society, vol. A234, 221 (1956).

(2) C. S. Barrett, Structure of Metals, McGraw-Hill, New York, 267 (1952).

and on the critical shear stress in each system. It has been found that the shear stress necessary to cause slip is the same for all three systems, (1) so that the slip system which operates depends on the orientation only. Therefore, by calculating the orientation factor with the maximum value for each crystal, and depending on the validity of the assumption made previously, it is possible to determine the resolved shear stress.

The orientation factors for the niobium (columbium) single crystals are recorded in Table 3, while the values for the corresponding resolved shear stresses at 0.05% plastic strain are recorded in Table 4.

Results of Tensile Tests

Tensile Testing Scheme

Table 5 shows the temperatures at which the different types of specimens were tested.

The results of these tests are shown in Figures 5 and 7 through 11. Observed results are reported and discussed in the following paragraphs.

Twinning

Twinning at low temperatures has been reported in most b.c.c. metals. Christian and Masters (2) have related this tendency to purity in their work with niobium (columbium).

(1) Allen et al., op. cit.

(2) J. W. Christian and B. C. Masters, private communication.

They have determined that the purer specimens of niobium (columbium) twin more readily than the impure. Biggs and Pratt, (1) on the other hand, have proposed that the nucleation of twinning is brought about by a stress concentration at the head of a rapidly piled-up array of dislocations produced by a burst of slip as a Frank-Read source is released from its atmosphere. They state further that pinning has less effect in niobium (columbium) than in other b.c.c. metals, so the nucleation of twins is more difficult. This would indicate that purer niobium (columbium) would twin less easily than the impure material. The experimental observations of Adams, et al. (2) on twinning at 20°K in niobium (columbium) support this proposal in that they found a small amount of slip with large twin bursts followed by a preponderance of slip and few twin bursts as deformation continued. This was attributed to the fact that twins, once formed, acted as barriers which allowed further dislocation pile-ups and twin nucleation. As deformation continued, most Frank-Read sources were released and twin nucleation gradually stopped.

In the present work, twinning was apparent only in tests at 4.2°K. It manifested itself by audible clicks and by sharp load reductions on the Instron chart. Though only one test was made with one zone pass material at 4.2°K, as

(1) W. D. Biggs and P. L. Pratt, Acta Metallurgica, vol. 6, 694 (1958).

(2) Adams et al., op. cit.

opposed to six tests with purer six pass material, the impure material exhibited a smaller tendency toward twinning. There were fewer bursts, and they were accompanied by much smaller load drops. This is in agreement with the findings of Christian and Masters. The twinning process in the present tests behaved as described previously in that the tendency decreased as the plastic portion of the stress-strain curve was approached. It then gave away completely to slip.

Slip Lines

Maddin and Chen (1) state that slip lines:

- (a) become broader with increasing deformation temperature;
- (b) are wavy if many slip planes are operating at the same time;
- (c) are localized if deformation is not uniform.

For niobium (columbium) in particular they observed:

- (a) lines which were straight and sharp;
- (b) bands having definite width which were branched or forked, yet seemed to be made up of straight lines.

In the present work, all tensile specimens were examined under optical microscope (magnification 120X)

(1) R. Maddin and N. K. Chen, Progress in Metal Physics, vol. 5, 53 (1954).

after testing. Slip lines were observed to be very wavy and curved. Some of the specimens which fractured at higher temperatures showed double lines which appeared to be bands. On the specimens which fractured at low temperatures, only a very few lines could be seen. These results indicated the probability that:

- (a) many slip planes were operating at the same time;
- (b) formation of bands was due to relatively high testing temperature;
- (c) slip lines disappeared at relatively low temperatures, at least when viewed under the optical microscope, because of the low temperatures.

Yield Point Phenomena

The appearance of yield points in b.c.c. metals is believed by many to be a function of impurity content, where the yield point appears as a result of impurity-locked dislocations being suddenly unlocked. Other investigators believe that this phenomenon is a function of grain size, where grain boundaries prevent the free movement of dislocations.

In the present work, no yield points were observed in either polycrystalline or single crystal specimens, or in the one one zone pass single crystals at any temperatures. Johnson (1) found no yield points at any temperature in his

(1) A. A. Johnson, Acta Metallurgica, vol. 8, 737 (1960).

work with niobium (columbium), whereas Adams et al. (1) observed yield points in the majority of their tests. An approximate comparison of the impurity levels in these tests shows:

Present six pass material - 99.7% pure

Johnson's niobium (columbium) - 99.6% pure

Present one pass material - 99.4% pure

Adams' et al. niobium (columbium) - 99.3% pure

This comparison indicates that impurities play the major role in determining whether or not a b.c.c. metal exhibits yield points as a part of its mechanical behavior.

Fracture

All specimens tested above 4.2°K parted in a ductile, knife-edge mode of fracture. Those tested in liquid helium showed brittle shear failures.

Ductility

In b.c.c. metals such as α -iron (2) and molybdenum (3), the method used for measuring the ductility has been to compare the uniform strains at the various testing temperatures. This indicates the ability of the material to pass on deformation from one local area to the next. As can be seen from Figures 7 and 8, niobium (columbium) exhibits little uniform strain while still possessing

(1) Adams et al., op. cit.

(2) R. L. Smith and J. L. Rutherford, AIME Transactions, vol. 209, 857 (1957).

(3) Lawley et al., op. cit.

ductility. Thus, uniform strain is not considered a satisfactory criterion for measuring ductility where niobium (columbium) is concerned. Instead, the effect of temperature on the reduction in area is used. In Figure 9, the results of Wessel, France, and Begley (1) and Wessell and Lawthers (2) are shown. In the former, fine-grained niobium (columbium) (110 grains/mm²) of purity comparable to that used in the present work was tested in tension. In the latter, slightly less pure material of grain size comparable to that in the present work was used. Their results show that the ductile to brittle transition takes place sharply at some point within a temperature range of 60 to 160°K. The exact temperature within this range seems to be a function of grain size and impurity content. These conclusions are in agreement with the results of several other workers as compared in the literature by Bechtold, Wessel, and France. (3)

Due to the testing scheme used in the present work, only four polycrystalline specimens were tested at single temperatures, the rest being used for reversible flow tests. The reduction in area which occurred in these specimens is plotted with the other results in Figure 9. As might be expected, they lie between the two curves of Wessel and

(1) E. T. Wessel, L. L. France, and R. T. Begley, Columbium Metallurgy, Interscience Publishers, New York, 459 (1961).

(2) E. T. Wessel, D. D. Lawthers, The Technology of Columbium, John Wiley and Sons, New York, 66 (1958).

(3) J. H. Bechtold, E. T. Wessel, and L. L. France, Refractory Metals and Alloys, Interscience Publishers, New York, 25 (1961).

co-workers, since they are intermediate in grain size and purity. Single crystal specimens fractured at these temperatures exhibited, within 3%, the same reduction in area, suggesting that the effect of the grain size on ductility might have an upper limit near the grain size used in the present work. In all interrupted temperature tests in which a test temperature of 77°K was used, reduction in area values were as much as 20% less than that recorded for the higher temperatures. This would seem to indicate a substantial drop in reduction in area for a testing temperature of 77°K. The only area of disagreement with other results occurs at the lowest temperature (4.2°K) where only one specimen, the least pure, fractured without measurable reduction in area. All others showed from 1 to 3% reduction. This difference might be a result of a difference in strain rate or of the type of impurities present in this material. In any case, in view of the limited number of specimens available and the possibility of errors in measurement, no conclusions are drawn from the figures on reduction in area except that they are in general agreement with those of previous workers.

Temperature Dependence of the Flow Stress

The flow stress has been defined as the applied stress at a particular value of strain required to continue deformation. (1) As mentioned previously, it has been

(1) Conrad and Schoeck, op. cit.

found to be dependent on the structure of the material. In order to make the flow stress dependent on temperature only, it is necessary to use the parameter $\Delta\sigma_{f1}$, the change in flow stress. For b.c.c. metals, this has been found to be independent of grain size and previous strain. (1, 2, 3) To obtain the values for the change in flow stress, the applied stress at each testing temperature is compared with that at a standard reference temperature (273°K) and the difference is plotted against temperature. In this way, any portion of the flow stress which results from the structure or previous mechanical treatment of the material will be eliminated.

The values for change in flow stress found in the present work have been recorded in Table 6. Both polycrystalline and single crystal six pass specimens were used, the values of applied stress for the polycrystalline specimens having been divided by two. Less pure one and three pass zone melted single crystals were also used. The values obtained were then plotted against temperature in Figure 10. The results show a marked increase in the temperature dependence of the flow stress at lower temperatures. This factor is in agreement with the experimental findings

(1) Adams et al., op. cit.

(2) Van den Syde, op. cit.

(3) Smith and Rutherford, op. cit.

on other b.c.c. metals. However, a definite, though slight amount of impurity dependence is exhibited, even at the lower temperatures. Christian and Masters (1) in their work with niobium (columbium), were unable to detect any such impurity dependence in materials ranging from slightly more pure to less pure than that used in the present work. Though chemical analyses are not considered completely accurate at the low impurity levels being dealt with here, it is felt that the type of impurities which are present may explain the difference in results. The impurity dependence found in the present work is less than that found by Lawley, Van den Sype, and Maddin (2) in their work with molybdenum. This result seems justified in view of the findings of Adams, Roberts, and Smallman (3) that the dislocation locking term for niobium (columbium) is approximately one-twentieth of the value found for molybdenum.

Temperature Dependence of the Yield Stress

The temperature dependence of the yield stress in b.c.c. metals is believed to be due either to the action of impurities in blocking the movement of dislocations or to frictional stresses inherent in the lattice which hinder the movement of free dislocations. The lack of any grain size dependence in the yield strength of niobium (columbium)

(1) Christian and Masters, op. cit.

(2) Lawley et al., op. cit.

(3) Adams et al., op. cit.

polycrystals has been attributed to the small value of the dislocation locking term (1) mentioned previously. This fact is further substantiated by the absence of discontinuous yield points in the higher purity levels of niobium (columbium), since this type of yield point is thought to be due to blocking of dislocation motion, either by impurity locking or pile-ups at grain boundaries in polycrystalline material. In view of these facts, it would seem that the temperature dependence of the yield stress is not a result of impurities but of a lattice friction force.

In this investigation, the yield stress at 0.05% plastic strain has been recorded for polycrystals in Table 7, and for single crystals as the resolved shear stress in Table 4. These values, when plotted against temperature, gave the curves pictured in Figures 5 and 11. Figure 11 also contains curves from other investigations which resulted from tests at comparable strains and strain rates. A comparison of these curves shows roughly identical shapes, hence, similar temperature dependence. The displacement between the curves is due to impurities, since the lower curve in Figure 11 is for the purest material, and the impurity level rises as the curves rise. The similarity in the shape of the curves would seem to indicate that the same mechanism was responsible for the temperature dependence of the yield stress no matter what the impurity level. The effect of this mechanism would then be enhanced by the action of greater amounts of impurities. If the impurity

(1) Ibid.

level reached a certain value which is dependent on the structure of the metal, dislocation locking would result, followed by a break in the locking at a higher stress and the presence of discontinuous yield points. Since the flow stress pictured in Figure 5 behaves in the same manner as the yield stress, the same argument is advanced for its action except that increased impurity content results finally in the ductile-to-brittle transition.

The mechanism which actually causes the temperature dependence of the yield and flow stresses in b.c.c. metals has yet to be determined. Most workers favor the Peierls-Nabarro force because it fits the experimental evidence and the condition that the mechanism be a frictional force inherent in the lattice. Whatever the mechanism might be, however, it seems clear from the results of the present work that it is inherent in the structure of the metal and that impurities act to enhance its resistance to dislocation motion.

CONCLUSIONS

1. Even at relatively high (2mm/min.) speeds of zone travel, a significant amount of zone refining takes place in niobium (columbium).
2. The impurity level determines whether niobium (columbium) exhibits discontinuous yield points when subjected to tensile testing.
3. The type of impurities present in niobium (columbium) determines whether the flow stress is impurity sensitive.
4. The temperature dependence of yield and flow stress in niobium (columbium) is due to a mechanism inherent in the material, such as the Peierls-Nabarro force, and the effects of this mechanism are increased by raising the impurity level.

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TABLE 1
ANALYSES OF ZONE MELTED NIOBIUM (COLUMBIUM)

Spectrographic Analysis (impurities in parts per million)			
<u>Element</u>	<u>As-Received</u>	<u>One Zone Pass</u>	<u>Six Zone Passes</u>
Iron	10	ND* < 10	ND < 10
Nickel	50	50	25
Chromium	ND < 5	ND < 5	ND < 5
Aluminum	25	5	5
Tin	ND < 10	ND < 10	ND < 10
Cobalt	ND < 25	ND < 25	ND < 25
Manganese	ND < 5	ND < 5	ND < 5
Magnesium	5	5	5
Silicon	10	10	10
Vanadium	ND < 25	ND < 25	ND < 25
Tantalum	1000	1000	1000
Boron	ND < 5	ND < 5	ND < 5
Titanium	ND < 50	ND < 50	ND < 50
Zirconium	5000	5000	5000
<u>Gas Analyses</u>			
Oxygen	460	84	7
Nitrogen (Kjeldahl)	82	38	25
<u>Carbon Analysis</u>			
Carbon	51	35	33

*ND < signifies not detected, less than

TABLE 2
RESISTIVITY OF ZONE MELTED NIOBIUM (COLUMBIUM)

	<u>Temp.</u> <u>(°K)</u>	<u>STD</u> <u>Voltage</u>	<u>STD</u> <u>Resistance</u>	<u>Current</u> <u>(amps.)</u>	<u>Specimen</u> <u>Voltage</u>
As- Received	273	.95525	10	.095525	.000428
	4.2	.3623	1	.3623	.000015
One Pass	273	.31850	1	.31850	.002030
	4.2	.44200	1	.44200	.0001774
Six Passes	273	.80115	1	.80115	.006597
	4.2	.79152	1	.79152	.0000012
Six Passes Lower end	273	.77014	1	.77014	.006402
	4.2	.78271	1	.78271	.0000041

TABLE 2--Continued

<u>Resistance</u> <u>ohm</u>	<u>A(10⁻⁴in)</u>	<u>L(in)</u>	<u>(micro-ohm)</u> <u>-cm</u>	<u>4.2/</u> <u>273</u>	<u>273/</u> <u>4.2</u>
.00448	31.192	2.532	14.2789	---	---
.00414	31.192	2.532	.12946	.00907	110.30
.006373	22.062	2.532	14.1044	---	---
.000401	22.062	2.532	.08875	.00629	158.92
.008234	16.619	2.532	13.72692	---	---
.0000015	16.619	2.532	.002517	.000133	5453.7
.008348	16.619	2.532	13.89104	---	---
.0000052	16.619	2.532	.008741	.000629	1589.2

TABLE 3
ORIENTATION FACTORS OF SINGLE CRYSTALS

<u>Specimen</u>	<u>Orientation Factor</u>
R22-Sp1	0.477
R22-Sp2	0.479
R23-Sp1	0.459
R23-Sp2	0.480
R24-Sp1	0.491
R24-Sp2	0.490
R24-Sp3	0.492
R25-Sp1	0.487
R25-Sp2	0.486
R25-Sp3	0.488
R26-Sp1	0.500
R26-Sp2	0.499
R26-Sp3	0.502

TABLE 4
RESOLVED SHEAR STRESSES AT 0.05%

<u>Plastic Strain</u>		
<u>Specimen No.</u>	<u>Test Temperature (°K)</u>	<u>Shear Stress (p.s.i.)</u>
One Zone Pass		
R24-Sp1	273	9,700
	77	32,500
	194	14,550
	273	6,250
R24-Sp2	4.2	68,000
R24-Sp3	273	7,550
Three Zone Passes		
R23-Sp1	273	8,350
	77	28,100
R23-Sp2	273	7,600
	77	23,200
	192	11,000
Six Zone Passes		
R22-Sp1	273	6,050
	77	25,400
R22-Sp2	273	6,200
	77	26,800
	373	4,500
	273	5,500
R25-Sp1	4.2	48,000
R25-Sp2	273	7,100
R25-Sp3	4.2	58,000
R26-Sp1	194	15,100
R26-Sp2	4.2	60,500
R26-Sp3	4.2	58,000

TABLE 5
TENSILE TESTING SCHEME

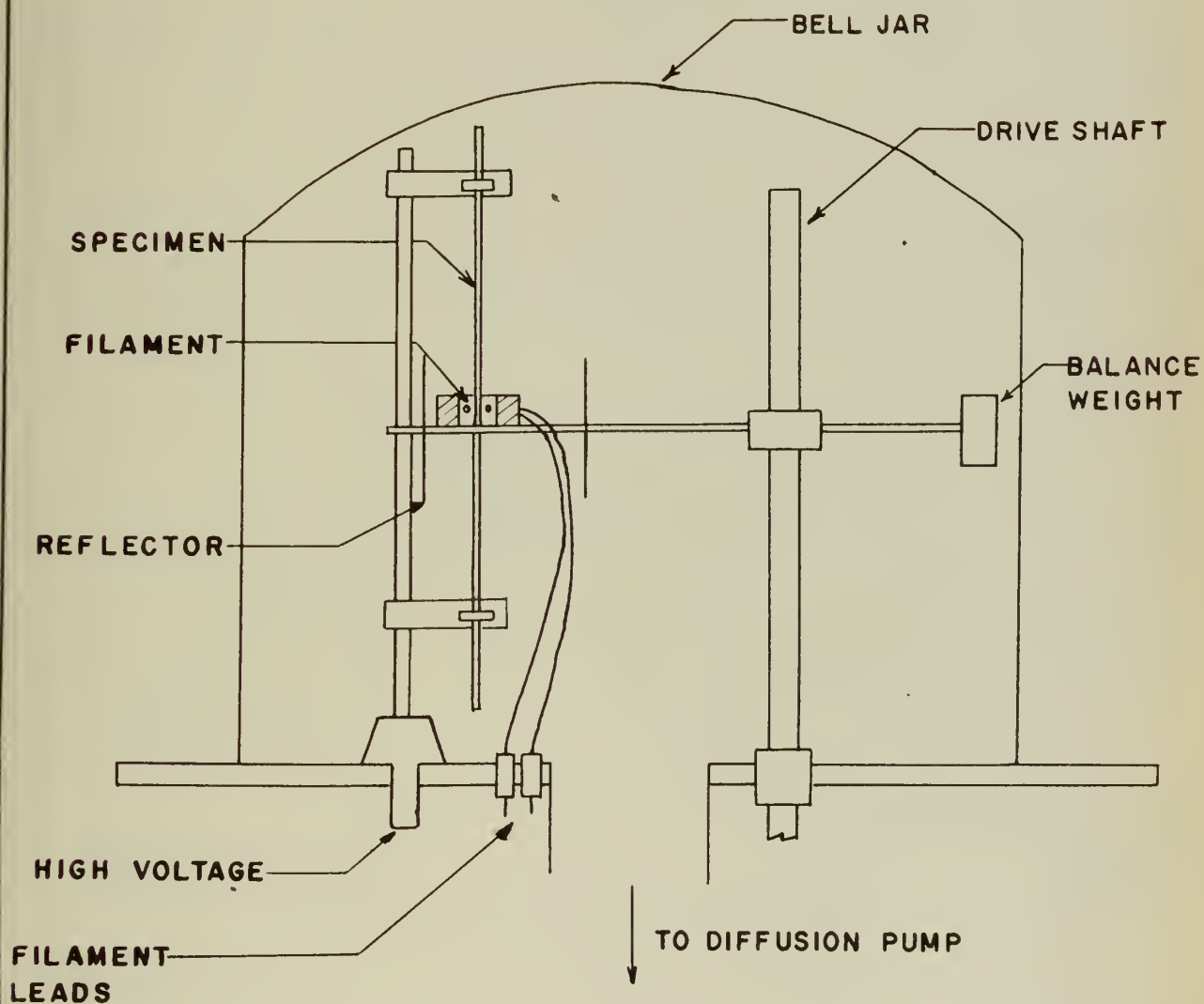
<u>Specimen No.</u>	<u>Temperature (°K)</u>	<u>Crystal Structure</u>
R19-Sp1	273	Polycrystal (Six Passes)
Sp2	196	
Sp3	273-77-373	
R21-Sp1	273-373-195-77	Polycrystal (Six Passes)
Sp2	273-373-77-196-273	
R22-Sp1	273-77	Single Crystal (Six Passes)
Sp2	273-77-373-273	
R23-Sp1	273-77	Single Crystal (Three Passes)
Sp2	273-77-192	
R24-Sp1	273-77-194-273	Single Crystal (One Pass)
Sp2	4.2	
Sp3	273	
R25-Sp1	4.2	Single Crystal (Six Passes)
Sp2	273	
Sp3	4.2	
R26-Sp1	194	Single Crystal (Six Passes)
Sp2	4.2	
Sp3	4.2	

TABLE 6
CHANGE IN FLOW STRESS

<u>Specimen</u>	<u>Temperature ($^{\circ}$K)</u>	<u>$\Delta\sigma_{f1}$ (p.s.i.)</u>
Six Zone Passes		
R21-Sp1	373	-1,750
	195	+3,750
	77	+19,200
R21-Sp2	373	-1,620
	77	+19,355
	196	+3,455
R22-Sp1	77	+19,350
R22-Sp2	77	+20,200
	373	-1,700
Three Zone Passes		
R23-Sp1	77	+20,750
R23-Sp2	77	+19,500
	194	+3,900
One Zone Pass		
R24-Sp1	77	+23,800
	194	+4,850

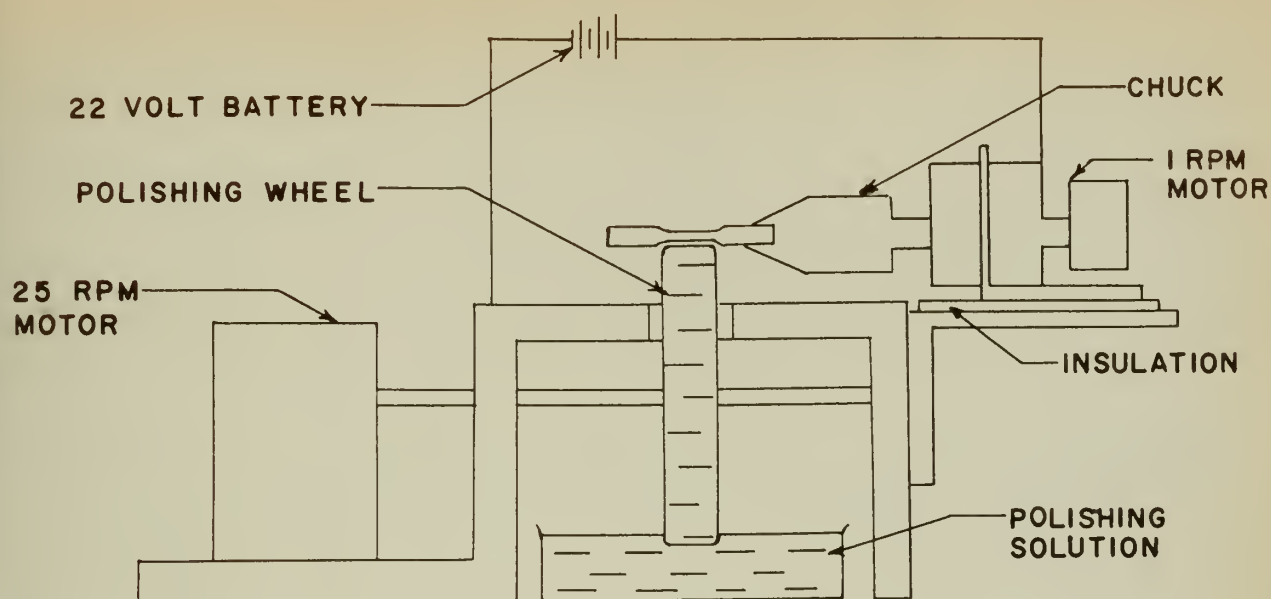
TABLE 7
POLYCRYSTALLINE YIELD STRESS AT
0.05% PLASTIC STRAIN

<u>Specimen No.</u>	<u>Temperature (°K)</u>	<u>Yield Stress (p.s.i.)</u>
R19-Sp1	273	11,200
Sp2	194	26,400
Sp3	77	56,700
	373	12,800
	273	14,600
R21-Sp1	273	14,100
	373	11,600
	195	21,600
	77	50,500
Sp2	273	15,640
	373	12,400
	77	54,350
	196	22,550
	273	12,250
Sp3	273	15,950
	77	55,400
	373	12,200
	273	17,275
R27-Sp1	4.2	126,700
Sp2	4.2	124,500

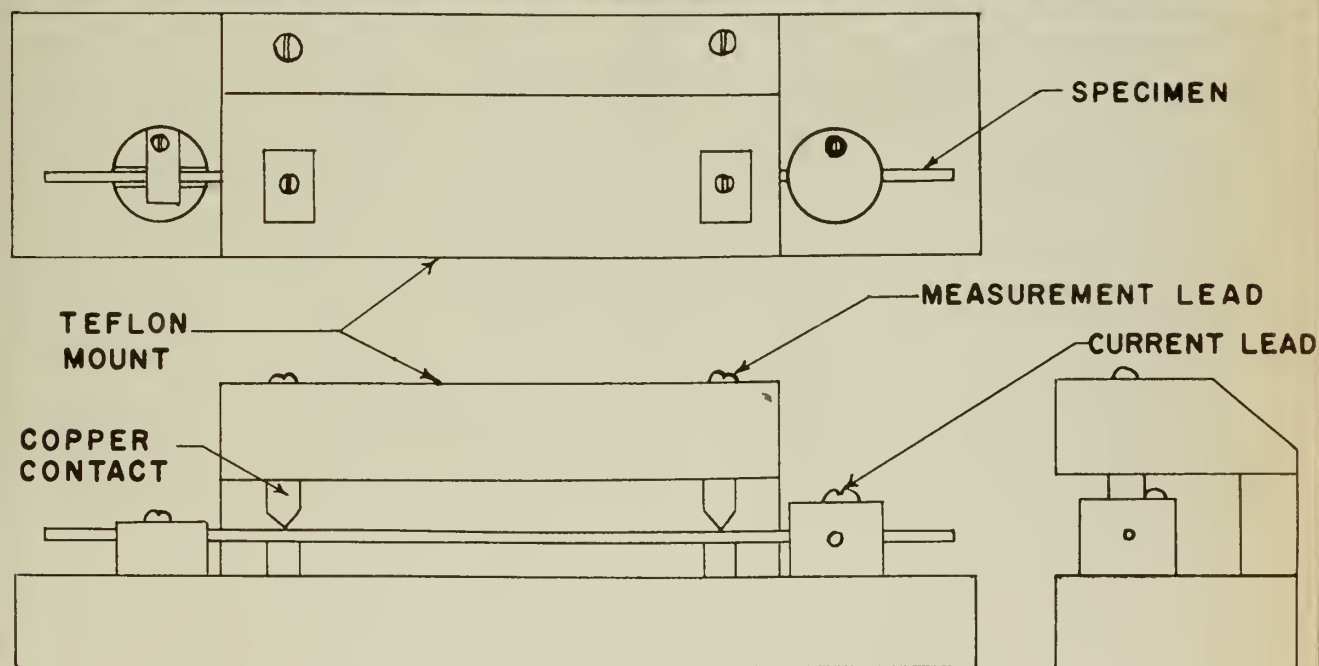


ZONE MELTING APPARATUS

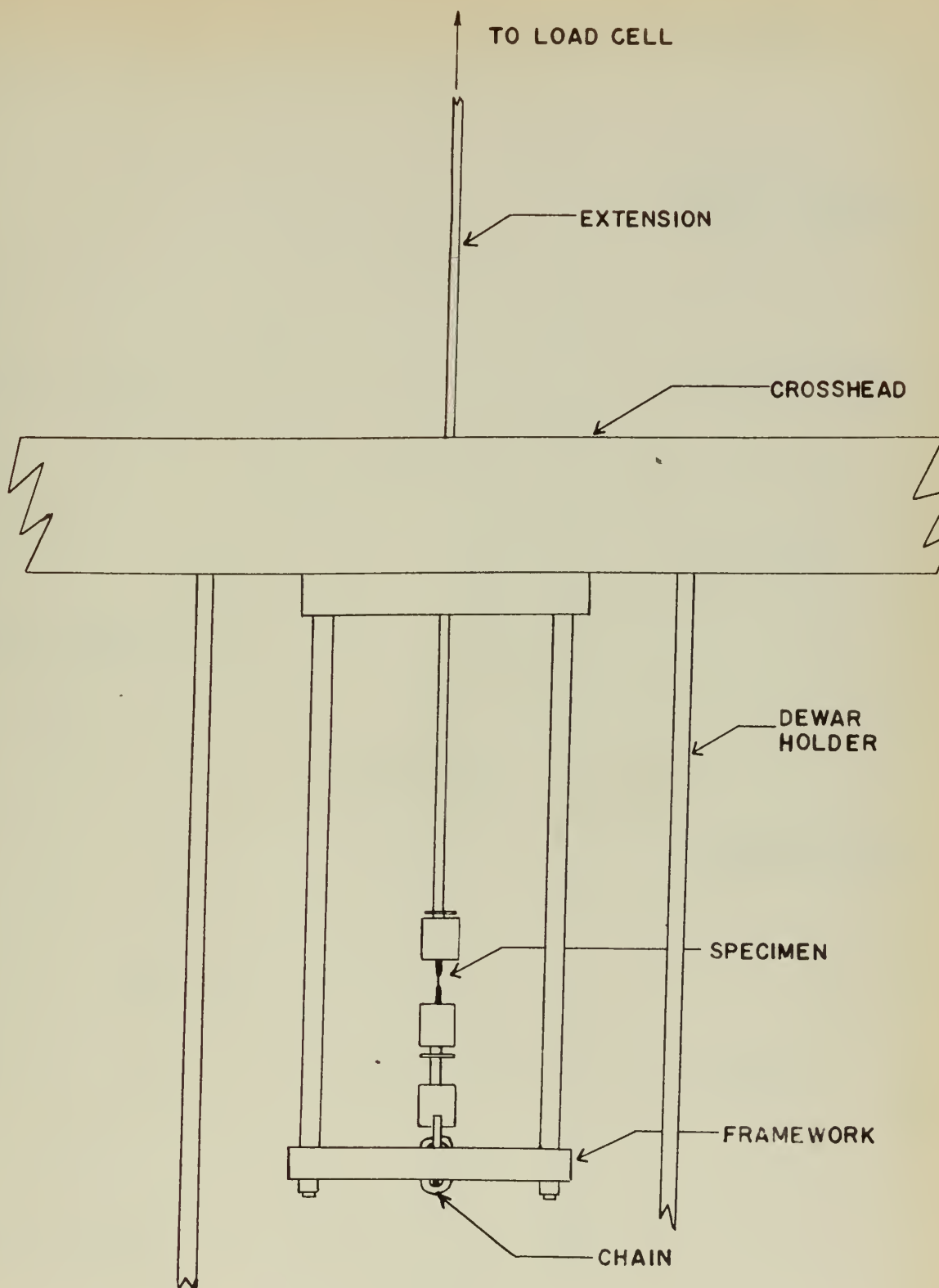
FIGURE 1



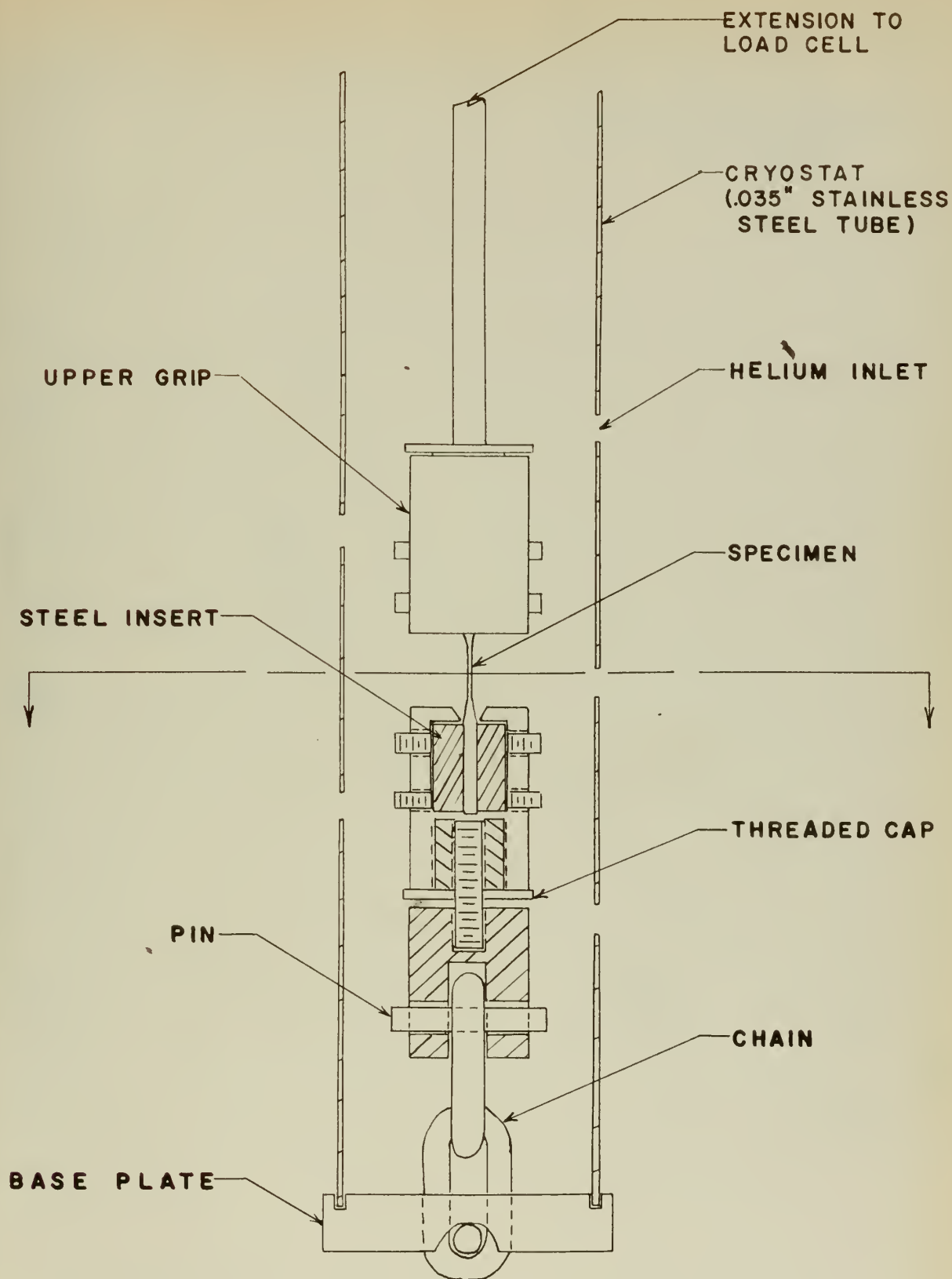
ACID LATHE
FIGURE 2a



RESISTIVITY MEASURING APPARATUS
FIGURE 2b



TENSILE APPARATUS
FIGURE 3



CRYOSTAT
FIGURE 4

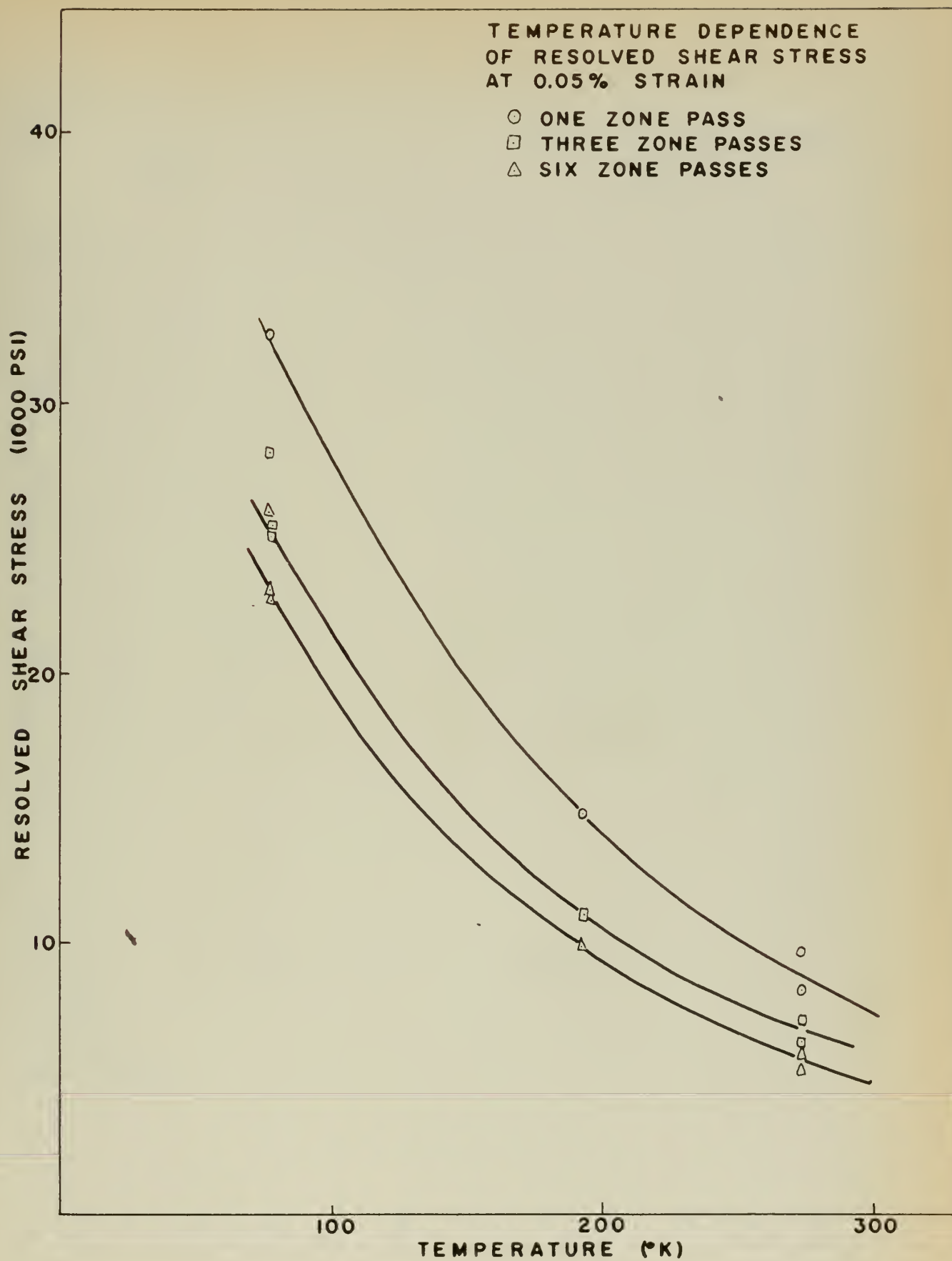


FIGURE 5

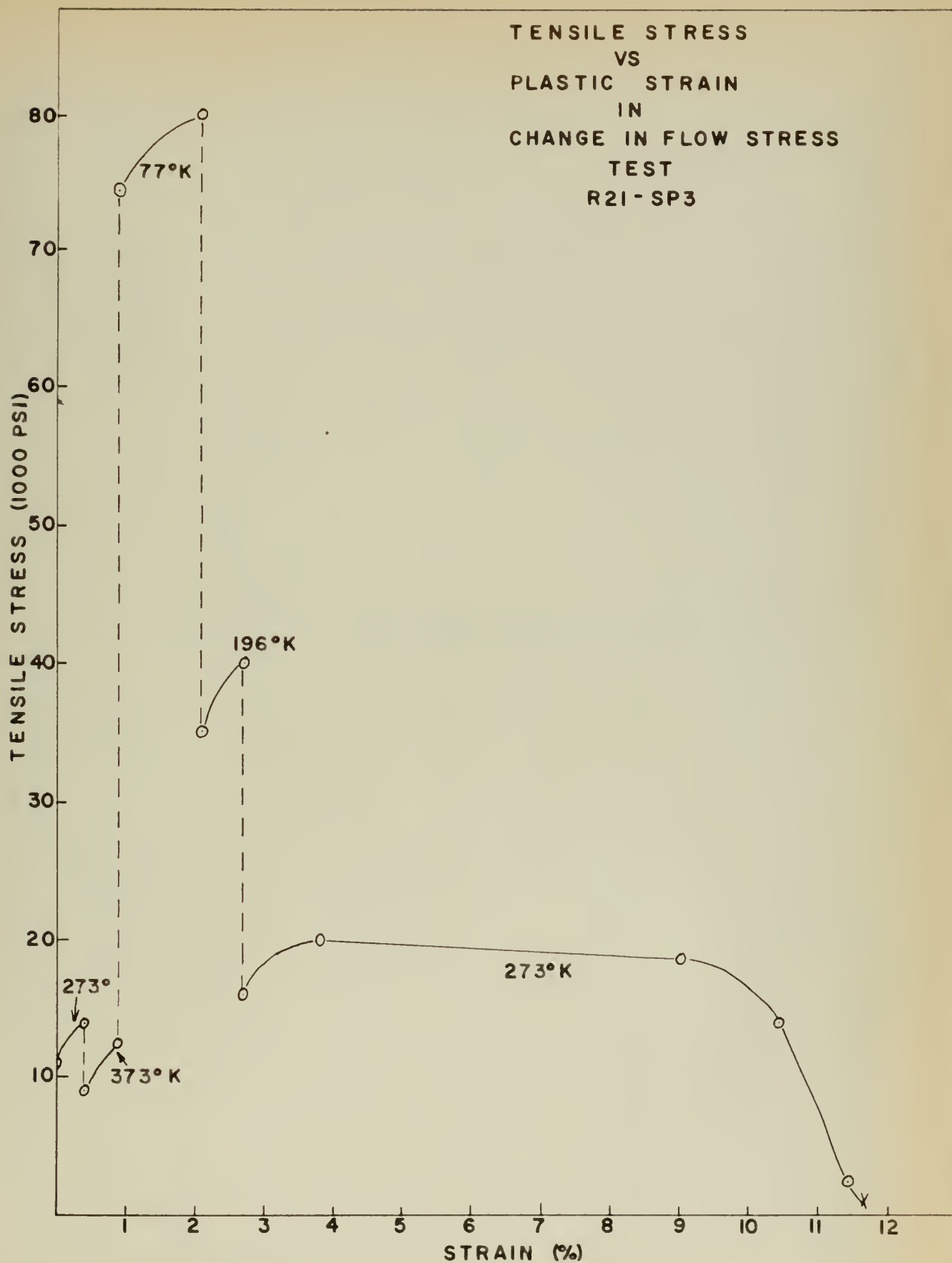


FIGURE 7

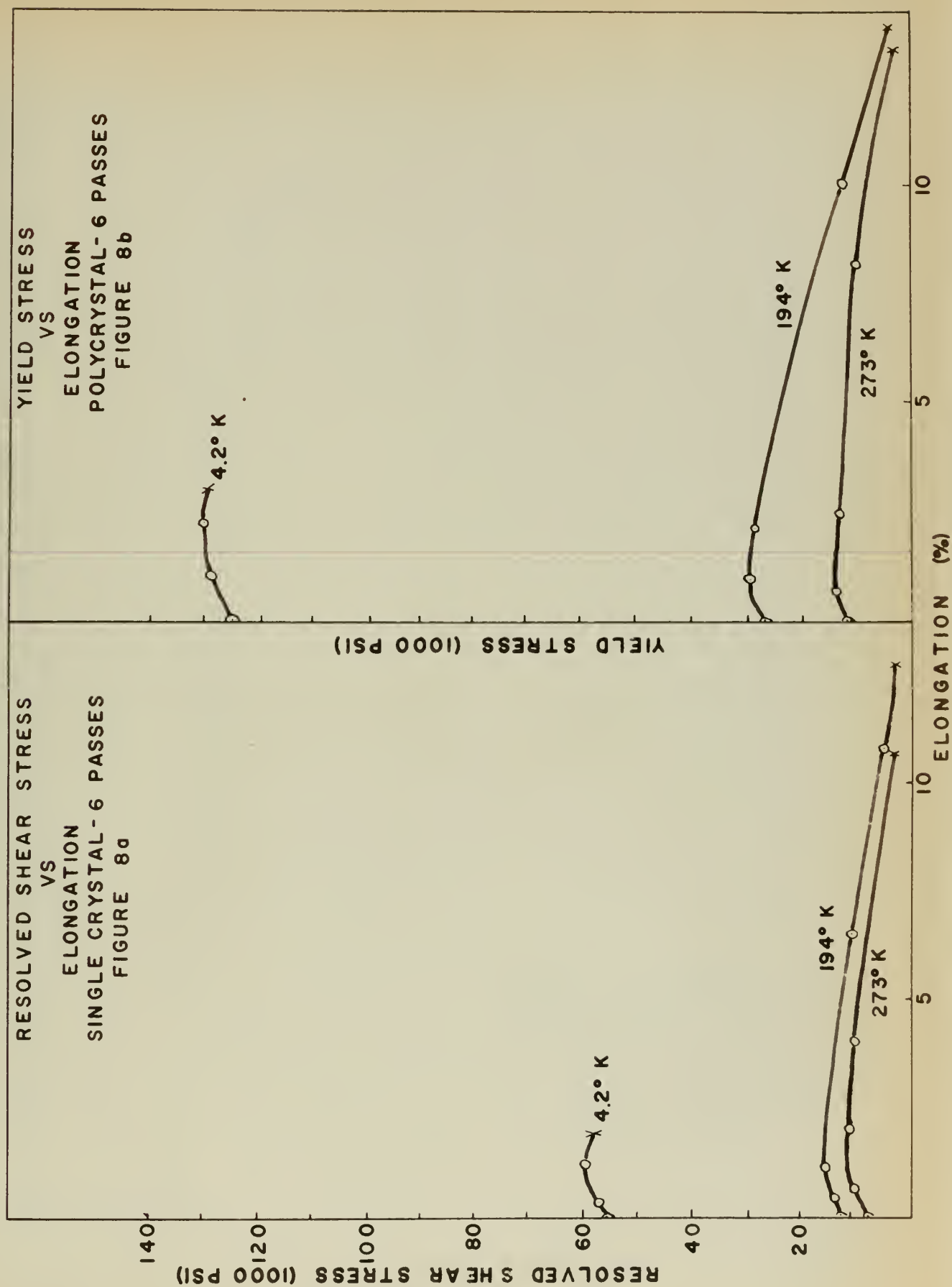


FIGURE 8

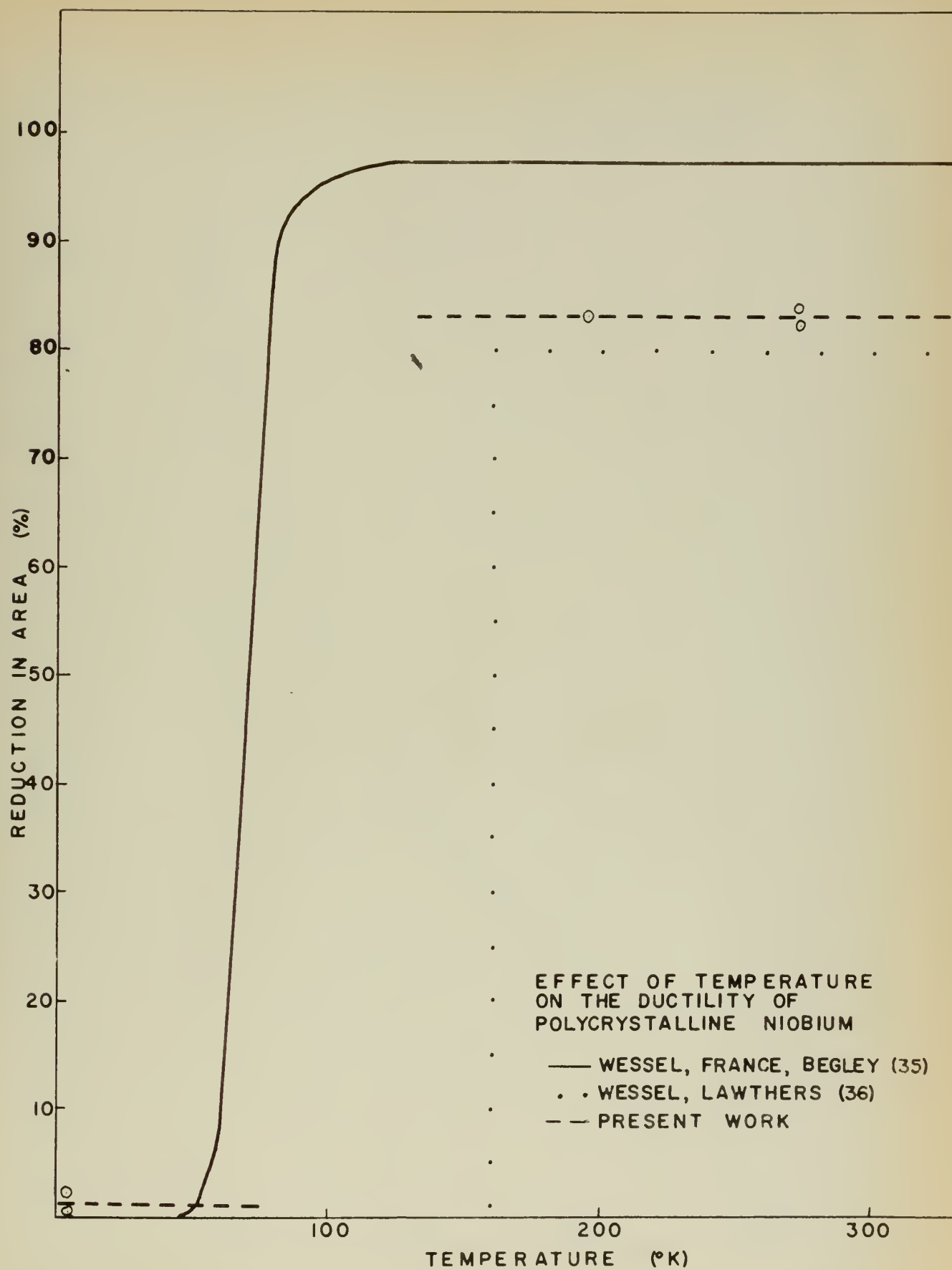


FIGURE 9

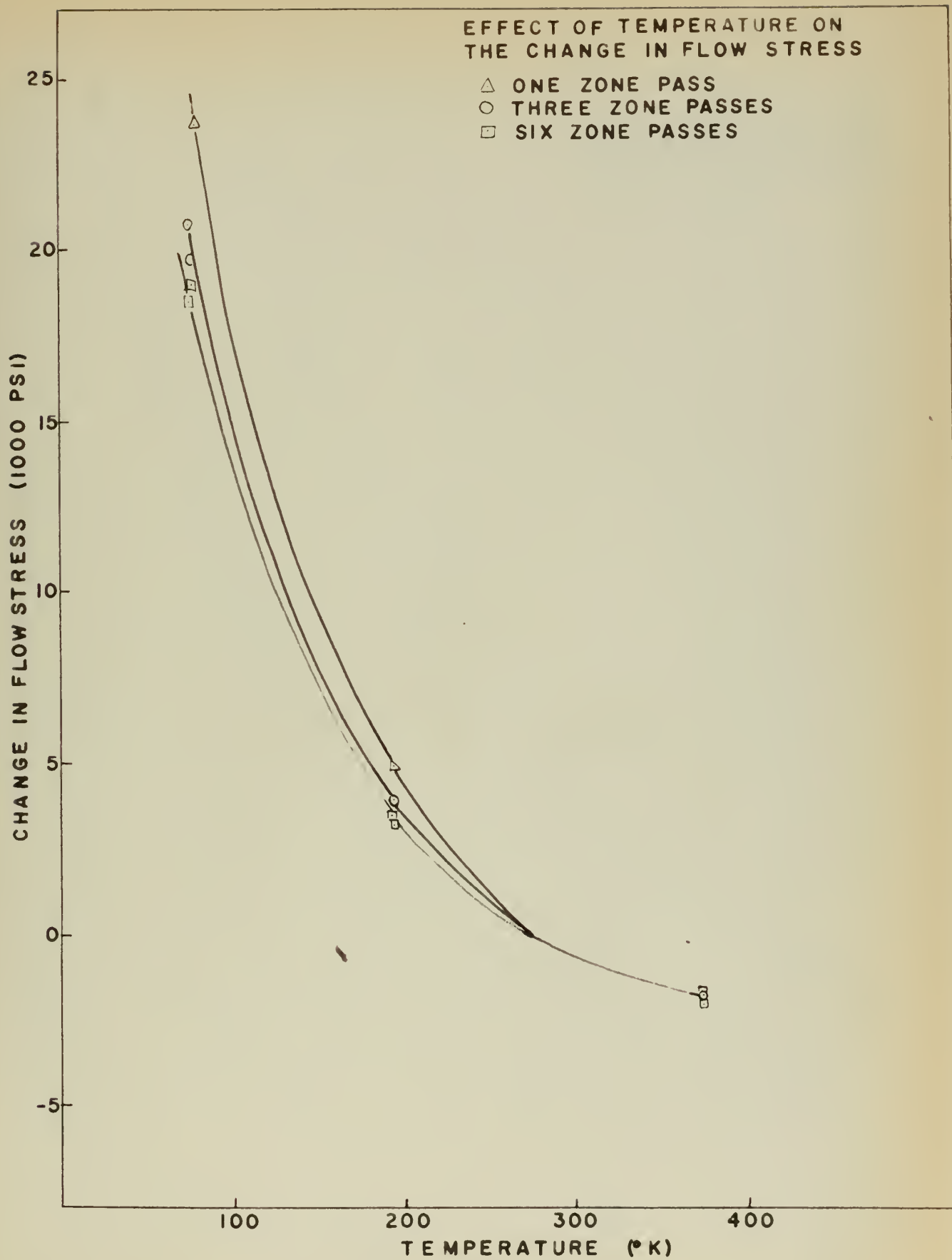


FIGURE 10

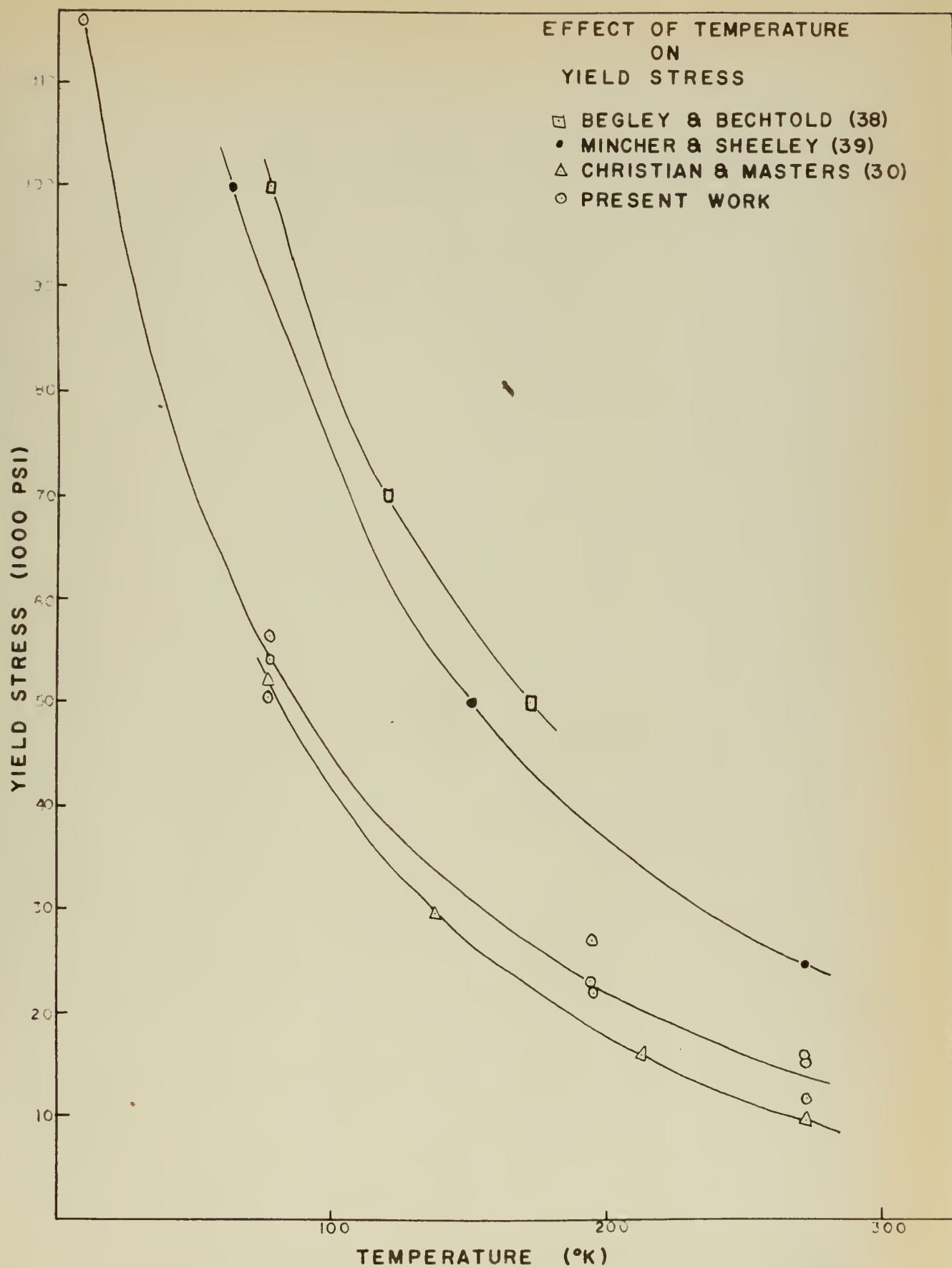


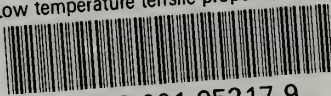
FIGURE II

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Low temperature tensile properties of zo



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